<u>Declaration under 37 CFR 1.132 with regard to US utility patent application number 10/675,138</u>

- I, Alan Reginald Minihan, a British subject of 18 Green Lane, Wallasey, Merseyside, U.K. declare the following:
- 1. I hold the degree of D. Phil. in Chemistry from University of Oxford and the degree of Master of Arts from University of Oxford. I am a Chartered Chemist and a member of the Royal Society of Chemistry.
- 2. I am presently employed as Group Product Development Manager by Ineos Silicas Limited, Bank Quay, Warrington, UK and have worked for a total of 21 years on the chemistry and structure of inorganic chemicals for Unilever plc, Crosfield Ltd, and Ineos Silicas Ltd.
- 3. The research work detailed below was carried out as part of a joint BRITE (EU-sponsored) research project (no F14W-CT95-0016) between British Nuclear Fuels Ltd. (UK), Crosfield Ltd. (UK), IVO International (Finland), University of Helsinki (Finland) University of Salford (UK) between 1st January 1996 and 31st December 1998. The table of data, Table 26, annexed to this document is from the final report detailing the work carried out in the project.
- 4. Crosfield ltd. changed its name to Ineos Silicas ltd. on 13th March 2001.
- 5. Table 26, which is annexed to this document, shows the distribution coefficients (Kd) for various isotopes in acid solution for tungsten (W)-doped antimony silicates (WSS samples HMS18) in comparison to antimony silicate (HMS10) and titanium (Ti) doped antimony silicate (HMS19). Description of the preparation of the materials tested is also included in table 26.
- 6. From the comparative data presented in the table it can be seen that the tungsten doped antimony silicates give much higher values for Kd (e.g 702, 8182, 14251-18303 for HMS18a1; i.e. good extraction behaviour) in comparison to the Kd values obtained for the titanium-doped antimony silicate (22, 187, 1.06). It is believed that the test results indicated herein are representative of the testing program, even though there may be other tests, not included herein, that may have been conducted in the time frame of the program.
- 7. From these data it was concluded that Ti was an undesirable dopant for antimony silicate to be used for extraction of radioactive metals from acid solution and work on this dopant was not progressed.
- 8. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

09 May 2006

MP100274 A-US

Signature

Date 09 May 2006

Name

Alan Reginald Minihan

Table 26. Distribution coefficients (Kd) for W doped antimony silicates

			77					1		Т	T -	T	T	T_	T	
³⁷ Co Kd [ml/g] in 0.1 M HNO ₃	1959	1509-4282		1.06	285	2012	75	116	09		14251-18303	1320	251	48.5 (dissolves)	1762	859
⁸³ Sr Kd [ml/g] in 0.1 M HNO ₃	09961	35515-102700		187	363	2012	141	199	118		8182	8918	2489	282 (dissolves)	41382	3252
in 0.1 M HNO ₃	590	1354-3702		7.7	400	472	118	220	205		702	670	272	85.8 (dissolves)	1332	10652
XRD trace	Amorphous	Crystalline (as antimonic acid)		Amorphous	Crystalline, AMP?	Amorphous	Amorphous	Amorphous	Amorphous		Amorphous	Amorphous	Amorphous	Cryst. Unknown	Amorphous	Semicryst. SbSi
Preparation method	1% mixture at 77C, overnight	1% mixture at 77C overnight	000	at 60C, 1a	at 77C, 3 days		at 77C, 2 days	at 77C, overnight	-,-		1% mixture at 77C	let to dry at 77C	at 77C overnight	-	let to dry at 77C	20h at 77C
Starting Sb:Si:W ratio	I:I (weighed)	1:1 (0.1 M solutions)	1.5 %	1:1:0.61 (sol)	1:1:1 (weigh.)	1:1:0.2 (weigh)	1:1:1:1 (sol)	1:2.5:1.7 (sol)	1:2.5:0.5 (sol)		1:1:0.5 (weigh.)	1:1:0.5 (weigh.)	1:1:1 (weigh.)	1:1:2 (weigh.)	1:1:0.1 (weigh.)	1:1:1 (weigh.)
Starting materials	KSb(OH), TEOS, HNO,	SbCl, in 4 M HCl, Na ₂ Si ₃ O, (Fluka)	DIAM DIAM	HMS10+11Cl	HMS12 + NH4(MoO ₃) ₂	HMS12 + NH4(MoO ₃) ₂	HMS10 + NH ₄ (MoO ₃) ₂	HMS10 + NH ₄ (MoO ₃) ₂	HMS10 + NH ₄ (MoO ₃) ₂		HMS12 +Na ₂ WO ₄ *2H ₂ O	HMS12 +Na ₂ WO ₄ *2H ₂ O	HMS12 +Na ₂ WO ₄ *2H ₂ O	HMS12 +Na2,WO4*2H2O	HMS12 +Na2WO4*2H2O	HMS10 +Na, WO, *2H,O
Sample name	HMS12 (KSS)		TiSS	MASSIS	HMS17a1	HMS 17a2	HMS17c1		1763			9			P#	HMS18c1
	_	2	r.	٦	4	5	9	7	∞		£	4	5	9	7	∞

	4.0/	433	7510	C134: 2001 (mr.)) : :	\\?	+Na2WO4*2H2O		
7		367	3142	Cryst ShSi (int?)	1 d at 77C	1:2.5:1.7 (sol)	HMS10	HMS18c8	117
	727				•		+Na2WO4*2H2O		
T	280	\$221	6924	Cryst. SbSi	let to dry at 77C	1:2.5:1.7 (sol)	HMS10	HMS18c7d	16
	?	1		•			+Na2WO,*2H2O		
	1440	42151	18697	Cryst. SbSi		1:1:0.5 (weigh.)	HMS10	*92818MH	15
	•	1		•			+Na2WO,*2H2O		
T	1011	9492	7008	Cryst. SbSi	-6-	1:2.5:0.5 (sol)	HMS10	HMS18c5	14
				•			+Na2WO4*2H2O		
Τ	611	9651	15356	Cryst. SbSi	1 d at 77C	1:2.5:1 (sol)	HMS10	HMS18c4	13
	2			`	•		+Na2WO4*2H2O		
T	640	8075	20970	Cryst. SbSi	let to dry at 77C	0.5:1:1 (weigh.)	HMS10	HMS18c3d	12
	•			•		,	+Na2WO4*2H2O		
Τ	224	5637	17745	Cryst. SbSi	2 days at 77C	0.5:1:1 (weigh.)	HMS10	HMS18c3	E
				1		•	+Na2WO4*2H2O		
Τ	1515	35499	17188	Cryst. SbSi	let to dry at 77C	1:1:1 (weigh.)	HMS10	HMS18c2d	01
							+Na2WO4*2H2O		
Γ	1552	4608	14441	Cryst. SbSi	2 days at 77C	[1:1:1 (weigh.)	HMS10	HMS18c2	6